



# **KERNFORSCHUNGSANLAGE JÜLICH GmbH**

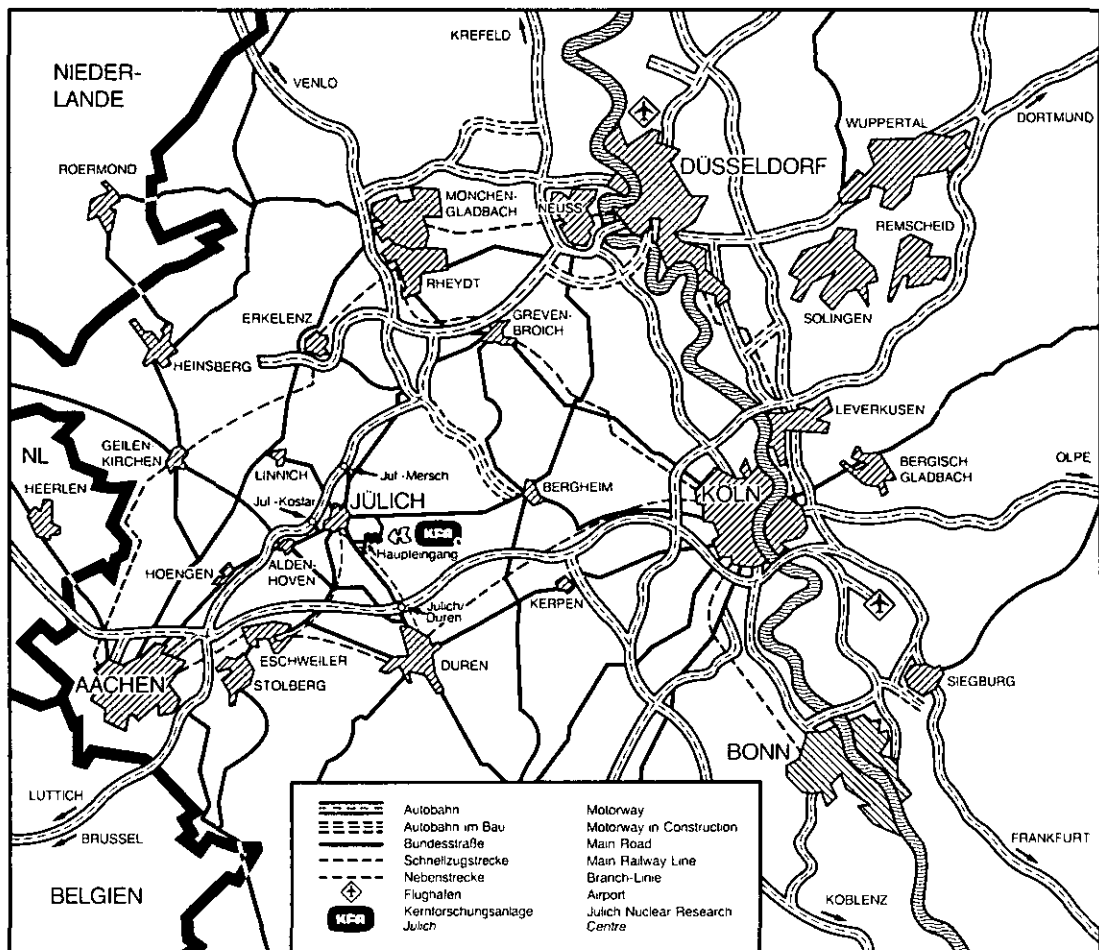
**Institut für Chemische Technologie  
der Nuklearen Entsorgung**

## **TEST OF THE PRECISION AND ACCURACY OF THE ISOTOPIC ANALYSIS OF HIGH ENRICHED URANIUM IN BISO-PARTICLES**

by

B.-G. Brodda, H. Kirchner, T. Görgenyi, F. Schinzer,  
E. Kuhn, A. Zoigner, E. Mainka, H. Deutsch, D. Thiele  
and E. Santner

**Jül - Spez - 251**  
**April 1984**  
ISSN 0343-7639



Als Manuskript gedruckt

## **Spezielle Berichte der Kernforschungsanlage Jülich – Nr. 251**

Institut für Chemische Technologie der Nuklearen Entsorgung Jül – Spez – 251

Zu beziehen durch: ZENTRALBIBLIOTHEK der Kernforschungsanlage Jülich GmbH

Postfach 1913 · D-5170 Jülich (Bundesrepublik Deutschland)

Telefon: 02461/610 · Telex: 833556-0 kf d

# **TEST OF THE PRECISION AND ACCURACY OF THE ISOTOPIC ANALYSIS OF HIGH ENRICHED URANIUM IN BISO-PARTICLES**

by

B.-G. Brodda, H. Kirchner  
Institut für Chemische Technologie der Nuklearen Entsorgung  
der Kernforschungsanlage Jülich GmbH, Jülich

T. Görgenyi, F. Schinzer  
NUKEM GmbH, Hanau

E. Kuhn, A. Zoigner  
Seibersdorf Analytical Laboratory  
International Atomic Energy Agency, Vienna, Austria

E. Mainka, H. Deutsch  
Institut für Radiochemie  
des Kernforschungszentrums Karlsruhe, Karlsruhe

D. Thiele, E. Santner  
Bundesanstalt für Materialprüfung, Berlin

# **Joint Programme**

on the  
**Technical Development and Further  
Improvement of IAEA Safeguards**  
between the Government of the  
Federal Republic of Germany and the  
International Atomic Energy Agency

TASK C.13

TEST OF THE PRECISION AND ACCURACY  
OF THE ISOTOPIC ANALYSIS OF HIGH ENRICHED URANIUM  
IN BISO PARTICLES

B.-G. BRODDA, H. KIRCHNER  
KERNFORSCHUNGSANLAGE JÜLICH GMBH, JÜLICH

T. GÖRGENYI, F. SCHINZER  
NUKEM GMBH, HANAU

E. KUHN, A. ZOIGNER  
INTERNATIONAL ATOMIC ENERGY AGENCY, VIENNA, AUSTRIA

E. MAINKA, H. DEUTSCH  
KERNFORSCHUNGSZENTRUM KARLSRUHE, KARLSRUHE

D. THIELE, E. SANTNER  
BUNDESANSTALT FÜR MATERIALPRÜFUNG, BERLIN

TEST OF THE PRECISION AND ACCURACY OF  
THE ISOTOPIC ANALYSIS OF  
HIGH ENRICHED URANIUM IN BISO-PARTICLES

B.-G. Brodda, H. Kirchner  
Institut für Chemische Technologie der Nuklearen Entsorgung  
der Kernforschungsanlage Jülich GmbH, Jülich

T. Görgenyi, F. Schinzer  
NUKEM GmbH, Hanau

E. Kuhn, A. Zoigner  
Seibersdorf Analytical Laboratory  
International Atomic Energy Agency, Vienna, Austria

E. Mainka, H. Deutsch  
Institut für Radiochemie  
des Kernforschungszentrums Karlsruhe, Karlsruhe

D. Thiele, E. Santner  
Bundesanstalt für Materialprüfung, Berlin

**FINAL REPORT**

**NOTICE**

This report was prepared as an account of work sponsored by the Government of the Federal Republic of Germany within the *Joint Programme on the Technical Development and Further Improvement of IAEA Safeguards between the Federal Republic of Germany and the IAEA*. Neither the authors of this report, nor the organization or industrial company they are affiliated with, nor the Federal Government assume any liability whatsoever for any use of this report or parts of it. Furthermore the content of this report does not reflect any policy of the Federal Government.

## 1. Introduction

The results of the isotopic analysis of high enriched uranium (HEU) in BISO-particles are routinely about 0.2 % low in the U-235 concentration compared to the nominal abundance of the uranium feed material. This is expected to be caused by natural uranium present in the thorium feed material and it was felt that the International Atomic Energy Agency needs to confirm the accuracy of its verification measurements on such fuels.

With the support of the Joint Programme on the Technical Development and Further Improvement of IAEA Safeguards between the Government of the Federal Republic of Germany and the International Atomic Energy Agency an interlaboratory experiment was performed to test the precision and accuracy of the isotopic analysis of HEU in pyro-carbon coated (Th,U) O<sub>2</sub> - particles (so called BISO - particles). This experiment was a follow-up to an international comparison of the chemical assay of uranium and thorium in THTR-fuel for safeguards purposes<sup>1, 2/</sup>.

The exercise was organized under the auspices of the Institut für Chemische Technologie der Nuklearen Entsorgung of the Kernforschungsanlage Jülich GmbH (KFA-ICT). The objectives and the outline of the experiment were established in a preparatory meeting held in Jülich in September 1982.

The fuel fabrication plant NUKEM in Hanau provided BISO-type particles, containing high enriched uranium, for the experiment and arranged for the distribution of the samples. The sample preparation was performed at NUKEM with the assistance of an IAEA staff member.

The samples were analyzed by mass-spectrometry in the following laboratories:

- Bundesanstalt für Materialprüfung (BAM), Berlin.
- Institut für Chemische Technologie der Nuklearen Entsorgung of KFA, Jülich.
- Institut für Radiochemie of Kernforschungszentrum, Karlsruhe
- NUKEM GmbH, Hanau
- Safeguards Analytical Laboratory of the IAEA, Seibersdorf.

The IAEA prepared a statistical evaluation of the analytical results.

The objective of the project was to explain observed differences and to define the state of the art of the U-235 isotope concentration determination in U-Th fuels and to estimate the magnitude of the error components in such analyses.

## 2. Description of the Experiment

### 2.1 General

In the design of the experiment it was considered that a potential source for biases in the measurement results could be a contamination with natural uranium. This contamination could originate from natural uranium contained in production thorium feed material or from the chemicals used in the sample preparation for mass spectrometry.

It was therefore decided to prepare three types of samples for the experiment:

- Type I: Samples prepared from BISO-particles, representative of normal production.
- Type II: Synthetic mixture prepared from SRM NBS-U-930 and from a  $\text{Th}(\text{NO}_3)_4$ -solution, containing 200 ppm natural uranium.
- Type III: Synthetic mixture prepared from SRM NBS-U-930 and from a  $\text{Th}(\text{NO}_3)_4$ -solution, containing a low concentration of natural uranium (analyzed as < 5 ppm by fluorimetry at NUKEM).

The sample preparation scheme, as described below, was aimed at producing synthetic mixtures (sample Types II and III) that are comparable in the heavy element contents and solution compositions to the Type I samples.

### 2.2 Starting materials

- 2.2.1: 8.7 g BISO-particles (~42.8 % Th, ~4.3 % U)
- 2.2.2: 0.61 g NBS-U-930 (in the form of  $\text{U}_3\text{O}_8$ )
- 2.2.3: 12.1 g  $\text{Th}(\text{NO}_3)_4 \cdot 4\text{H}_2\text{O}$  (high purity quality; additionally purified by two subsequent oxalate precipitations). The U-concentration was determined by fluorimetry as < 5 ppm.
- 2.2.4: Solution of a natural uranyl nitrate (0.5 mg U/ml).
- 2.2.5: THOREX-reagent (3,4,5) for the dissolutions (mixture of 13 M  $\text{HNO}_3$ /0.1 M  $\text{Al}(\text{NO}_3)_3$ /0.04 M NaF).

## 2.3 Sample preparation

### 2.3.1 Sample TYPE I

8.7251 g BISO-particles were ignited in air to burn-off the pyrocarbon-coating and dissolved in 22 ml THOREX-reagent. After complete dissolution the volume was adjusted to 50 ml with demineralized H<sub>2</sub>O and subsamples of 7 ml taken for each of the 5 laboratories (about 535 mg Th and 53 mg U per sample).

### 2.3.2 Sample TYPE III

0.61 mg of NBS-U-930 were dissolved in 10 ml THOREX-reagent, mixed with a solution obtained from dissolving 12.07 g Th(NO<sub>3</sub>)<sub>4</sub> · 4 H<sub>2</sub>O in 20 ml THOREX-reagent and finally diluted to 100 ml with demineralized H<sub>2</sub>O.

For each laboratory a 10 ml aliquot was taken from this solution (about 500 mg Th and 52 mg U per sample).

### 2.3.3 Sample TYPE II

To the remaining 50 ml of the TYPE III-mixture 1 ml of U-solution, containing 0.5 mg natural uranium, was added. Aliquots of 10 ml were taken for each of the 5 laboratories.

## 2.4 Reference values for the synthetic mixtures

### 2.4.1 Sample TYPE III

As the Th-nitrate used for this experiment was especially purified and its U-content verified as 5 ppm, the reference values given in the NBS-certificate can be applied:

$$\underline{1.0759 \pm 0.0020 \text{ wt\% U-234}}$$

$$\underline{93.276 \pm 0.010 \text{ wt\% U-235}}$$

$$\underline{0.2034 \pm 0.0006 \text{ wt\% U-236}}$$

$$\underline{5.445 \pm 0.005 \text{ wt\% U-238}}$$

(1 ppm of natural uranium in the Th-nitrate used would lower the reference value by 0.0022 wt. % U-235 to 93.274).



#### 2.4.2 Sample TYPE II

To 50 ml of the TYPE-III-solution 0.5 mg of natural uranium were added. The reference value for sample TYPE-II is calculated as:

93.097 wt% U-235

#### 2.5 Measurement scheme

Each laboratory received a set of three samples and was asked to follow for each sample type the scheme shown in Figure 1:

- Each sample was divided into two parts for independent separations.
- Per separated fraction 2 mass-spectrometric measurements were performed and reported on that form.

The separation procedures of the individual laboratories are compiled in Annex 1.

### 3. Results and Evaluation

#### 3.1 Results

The individual U-235 concentration results reported by the 5 laboratories for the 3 sample types are summarized in Table 1 in coded format. The individual results for the minor isotopes U-234 and U-236 are given in Annex 2.

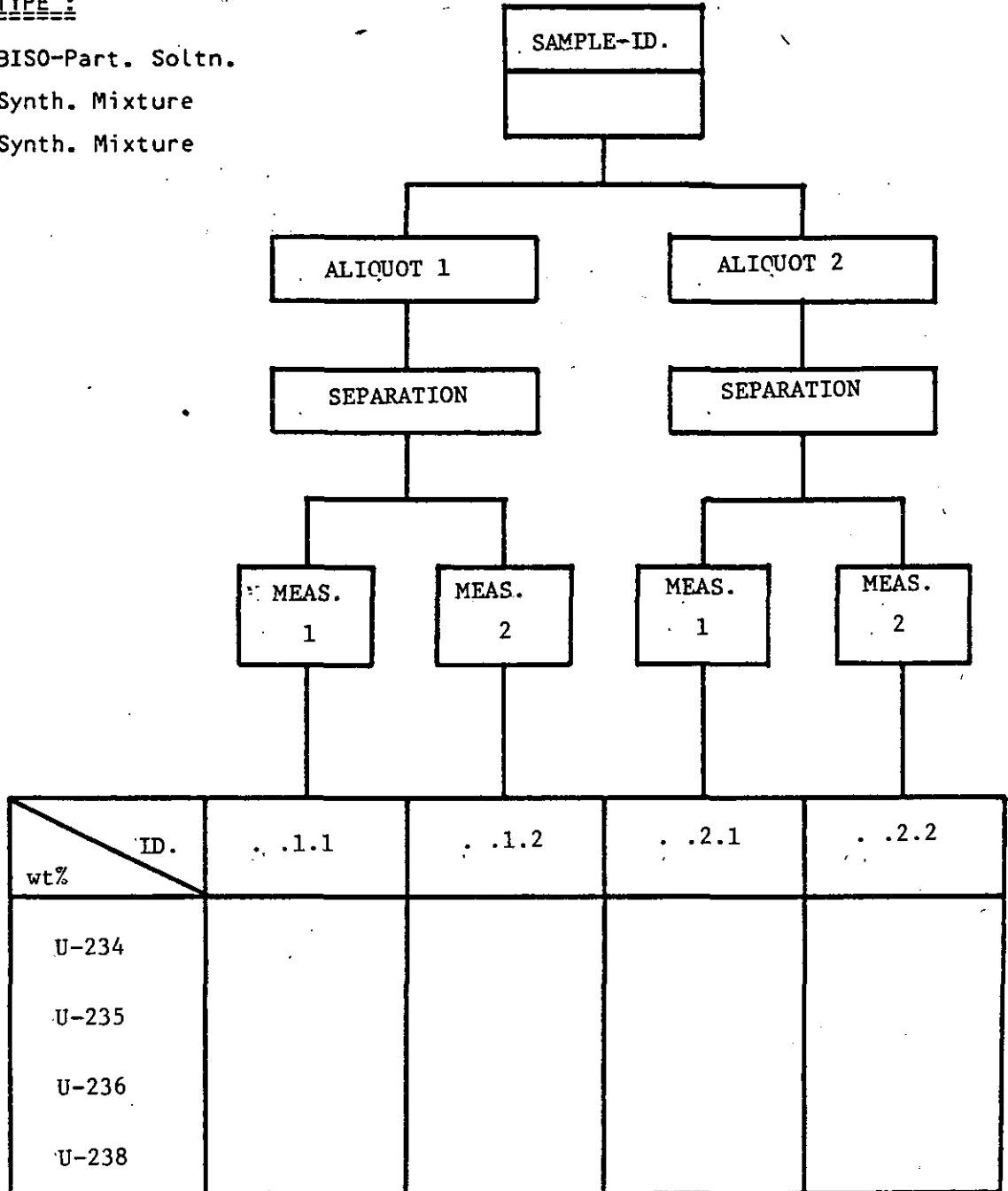
FIGURE 1

ANALYTICAL SCHEME AND REPORT FORM

LABORATORY:

SAMPLE TYPE\*:

- ☐ I: BISO-Part. Soltn.
- ☐ II: Synth. Mixture
- ☐ III: Synth. Mixture



\*Sample information: Volume..... 7 ml (Type I), 10 ml (Type II and III).  
 Medium.....THOREX-reagent  
 Thorium.....~500 mg total  
 Uranium.....~50 mg total  
 U-235.....~93 wt.%

Table 1  
Individual Analytical Results

Sample Type	Measurement	U-235 Concentration (wt%)				
		Lab. 1	Lab. 2	Lab. 3	Lab. 4	Lab. 5
I	..1.1	92.949	93.006	92.983	92.95	92.966
	..1.2	92.956	93.006	92.989	92.94	92.952
	..2.1	92.952	92.985	92.980	92.95	92.961
	..2.2	92.965	92.967	92.977	92.95	92.963
II	..1.1	93.091	93.100	93.103	93.08	93.092
	..1.2	93.087	93.103	93.107	93.09	93.093
	..2.1	93.088	93.099	93.108	93.09	93.096
	..2.2	93.093	93.093	93.111	93.10	93.094
III	..1.1	92.268	93.273	93.272	93.27	93.273
	..1.2	93.272	93.276	93.273	93.27	93.271
	..2.1	93.260	93.272	93.270	93.26	93.268
	..2.2	93.263	93.279	93.276	93.26	93.264

### 3.2 Evaluation Methodology

For each sample type, according to the measurement scheme (Figure 1) an individual measurement,  $x_{ijk}$ , is subject to 3 error terms. It can thus be described by the following model:

$$x_{ijk} = \mu + b_i + s_{ij} + t_{ijk}$$

where

- i identifies the laboratory
- j identifies the subsample
- k identifies the measurement on subsample j by laboratory i
- $\mu$  is the "true" value
- $b_i$  is the laboratory bias, including errors in the calibration of the method
- $s_{ij}$  is the treatment (separation error)
- $t_{ijk}$  is the measurement error.

For each sample type the results have been evaluated in two steps (a detailed description of the analyses of variance (ANOVA) has been given in the report on the Chemical Assay of (Th, U)<sub>2</sub> Coated Particles<sup>1/</sup> and is not repeated here).

### 3.3 Results of the individual laboratories

The results of each laboratory were submitted to a one-way analysis of variance. This statistical analysis yields estimates for

- the measurement errors, SI
- the separation errors, SH

These two components are considered in the computation of the standard error, SE.

An F-test allows to judge of the significance of variability between the subsample means when compared to the measurement error.

Table 2 summarizes the results of the one-way analyses of variance.

T a b l e 2  
Results of the 1-Way ANOVA (individual laboratories)

Sample Type	U-235 Concentration (wt%)				
	Lab. 1	Lab. 2	Lab. 3	Lab. 4	Lab. 5
I Measurement Error, SI	0.007	0.009	0.003	0.007	0.007
Separation Error, SH	neg.	0.020	0.005	neg.	neg.
Laboratory Mean, X	92.955	92.991	92.982	92.948	92.961
Standard Error, SE	0.004	0.015	0.004	0.004	0.004
F-test (sign. level of SH)	50	92	85	45	29
II* Measurement Error, SI	0.003	0.003	0.003	0.007	0.001
Separation Error, SH	neg.	0.003	0.003	0.005	0.002
Laboratory Mean, X	93.090	93.099	93.107	93.090	93.094
Standard Error, SE	0.002	0.003	0.002	0.005	0.001
F-test (sign. level of SH)	31	76	79	71	84
III** Measurement Error, SI	0.003	0.004	0.003	0.007	0.002
Separation Error, SH	0.006	neg.	neg.	0.005	0.004
Laboratory Mean, X	93.266	93.275	93.273	93.265	93.269
Standard Error, SE	0.004	0.002	0.002	0.005	0.003
F-test (sign. level of SH)	92	18	11	71	88

\* Reference Value: 93.097 wt. % U-235

\*\* Reference Value: 93.276 wt. % U-235

These results allow the following conclusions:

- The measurement errors estimated are in all cases smaller than 0.01 % rel. and demonstrate a high level of measurement precision.
- No significant separation error is detected for the individual data sets. This allows to conclude that the separation procedures applied do not introduce additional errors of analytical significance. In fact some of the estimates of  $SH^2$  gave negative values; this is possible in the cases where the measurement error variance is dominating.

### 3.4 Between laboratory comparison

For each sample type the analytical results of all the laboratories were submitted to a two-way analysis of variance. This statistical analysis yields estimates of the standard deviation components for:

- the measurement error, SIQ
- the mean separation error, SB
- the between laboratory error, SA.

The statistical significances of the variabilities between separations (groups) and between laboratories (classes) are tested by means of F-tests (FB and FA in Annex 3). The consistencies of the measurement errors of the individual laboratories and the fluctuations between separations are tested by means of Bartlett-tests (CHISQI and CHISQZ in the Annex).

The standard errors, associated with the means given in Table 3 have been calculated by the following equation

$$SET^2 = \frac{SA^2}{NL} + \frac{SB^2}{NS} + \frac{SIQ^2}{NM} \quad (df = NL-1),$$

where,      NL = No. of laboratories (5)  
              NS = No. of separations (10)  
              NM = No. of measurements (20)

Computer print-outs of the Analyses of Variance are given in Annex 3.

Table 3  
Results of 2-Way ANOVA (all laboratories)

		Sample Type		
		I	II	III
Measurement Error, SIQ	wt%	0.007	0.004	0.004
	% rel.	0.008	0.004	0.004
Separation Error, SB	wt%	0.009	0.003	0.004
	% rel.	0.010	0.003	0.004
Between Laboratory Error, SA	wt%	0.017	0.007	0.003
	% rel.	0.018	0.007	0.003
Grand Mean	wt%	92.967	93.096	93.270
Standard Error, SET	wt%	0.008	0.003	0.002
	% rel.	0.009	0.004	0.002
<u>Statistical Significance (F-tests, 99 % P)</u>				
Variability between separations		No	No	No
Variability between laboratories		No	No	No
<u>Consistency of Errors (<math>\chi^2</math>-tests, 99 % P)</u>				
Laboratory measurement errors		Consistent	Consistent	Consistent
Fluctuations between separations		Consistent	Consistent	Consistent

The results shown in Table 3 allow the following conclusions for all 3 types of samples:

- The measurement precisions obtained by the 5 laboratories are consistent.
- The measurement errors are less than 0.01% relative.
- The separation step does not introduce statistically significant errors; they are estimated to be equal or lower than 0.01 % relative.
- The between laboratory errors are smaller than 0.02 % rel. and statistically not significant.
- In the case of sample TYPE-I, which is an unknown sample, the fluctuation between the laboratories (0.018 % rel.) can be considered as the fluctuation of the laboratory biases.

### 3.5 Comparison to reference values (samples TYPE II and III)

Reference values are available for the samples of TYPE-II and TYPE-III (synthetic mixtures). Therefore the actual magnitude of the laboratory biases can be calculated, as summarized in Table 4.

T a b l e 4  
Comparison to Reference Values

Sample Type	U-235 (wt%)			Difference (Ref.-Mean)	
	Ref. Value	Grand Mean	Std. Error	wt%	% rel.
II	93.097	93.096	0.0033	0.001	0.001
III	93.276	93.270	0.0019	0.006	0.006

Two t-tests indicate that for both synthetic mixtures the differences between the reference value and the grand mean are statistically not significant when compared to the standard error (99 % probability level).



#### 4. Conclusions

The experiment has shown that a high level of measurement performance can be achieved in the U-235 concentration determinations by mass-spectrometry for (Th, U)O<sub>2</sub>-BISO-type coated particles. The separation step in the sample preparation for mass spectrometry does not introduce significant additional errors.

The measurement, separation and between laboratory errors are all well below 0.02 % rel. and are satisfying safeguards requirements.

The experiment is not in disagreement with the conclusion that the differences observed in the U-235 concentration determinations of high enriched uranium in BISO-particles, when compared to the nominal abundance of the uranium feed material, are due to depletion by natural uranium in the thorium feed material.

#### 5. Acknowledgements

The authors are indebted to the head of the IAEA Safeguards Analytical Laboratory, Dr. S.Deron, for his valuable discussions and especially to the technical laboratory personnel for performing the high precision analytical work.

References

1. H. Aigner et. al., IAEA-Report, IAEA/RL/85 (Sept. 1981)
2. D. Thiele et. al., J. Nucl. Mater. 113 (1983) 142
3. H. Franz et. al., Fresenius Z. Anal. Chem. 292 (1978) 353
4. R. E. Blanco et al., USAEC-Report ORNL-3219 (1961)
5. L. Küchler et. al., Kerntechnik 12 (1970) 327

Annex 1

Short Description of the Separation Procedures

- Lab. 1: Thorium separated by oxalate precipitation. U/Al-solution used for mass spectrometry without further separation.
- Lab. 2: Thorium separated by oxalate precipitation. Extraction of uranium with ethyl ether from the filtrate (saturated with  $\text{Ca}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$ ).
- Lab. 3: Uranium separation by anion exchange (8M  $\text{HNO}_3$ ).
- Lab. 4: Thorium separated by oxalate precipitation. Extraction of uranium with ethyl acetate (using  $\text{Al}(\text{NO}_3)_3$  as salting out agent).
- Lab. 5: Uranium separation with extraction chromatography, using 100% TBP on Voltalef powder. Washing with 6M HCl, uranium elution with  $\text{H}_2\text{O}$ .

Annex 2

Individual Analytical Results: Minor Isotopes U-234 and U-236

Sample Type	Measurement	U-234 Concentration (wt%)					U-236 Concentration (wt%)				
		Lab.1	Lab.2	Lab.3	Lab.4	Lab.5	Lab.1	Lab.2	Lab.3	Lab.4	Lab.5
I	..1.1	0.937	0.950	0.930	0.90	0.931	0.414	0.384	0.409	0.43	0.412
	..1.2	0.932	0.950	0.930	0.92	0.933	0.413	0.385	0.408	0.42	0.408
	..2.1	0.931	0.932	0.928	0.92	0.931	0.413	0.411	0.409	0.43	0.410
	..2.2	0.933	0.932	0.929	0.92	0.930	0.412	0.421	0.409	0.43	0.411
II	..1.1	1.074	1.074	1.073	1.06	1.079	0.202	0.203	0.201	0.21	0.201
	..1.2	1.078	1.074	1.073	1.05	1.077	0.203	0.203	0.201	0.20	0.201
	..2.1	1.073	1.074	1.071	1.06	1.073	0.203	0.203	0.202	0.21	0.202
	..2.2	1.072	1.073	1.072	1.06	1.079	0.205	0.203	0.201	0.21	0.203
III	..1.1	1.075	1.076	1.075	1.05	1.076	0.203	0.204	0.202	0.21	0.201
	..1.2	1.073	1.076	1.073	1.06	1.077	0.203	0.204	0.201	0.21	0.205
	..2.1	1.081	1.075	1.075	1.06	1.077	0.205	0.203	0.202	0.21	0.201
	..2.2	1.081	1.075	1.076	1.06	1.075	0.206	0.204	0.204	0.21	0.202

SAMPLE TYPE T U-235 RESULTS

ANNEX 3 (page 1a)

LIST OF ITEMS; ONE-WAY VARIANCE ANALYSIS ON INDIVIDUAL SAMPLES

LINE NO.	SAMPLE CODE	CLASS NO.	GROUP NO.	ITEM NO.	ITEM VALUE	GROUP MEAN	STD.DEV.	CLASS MEAN	CLASS STD.DEV.	CLASS SZ	CLASS F	CLASS DF	CLASS CHISG
1	LAB. 1	1	1	1	0.92949E+02	0.92952E+02	0.92955E+02	0.92955E+02	0.60043E-02	0.66191E+00	0.66191E+00	0.23985E+00	1
2				2	0.92956E+02	0.49524E-02	0.69508E-02	0.69508E-02	0.00000E+00	1/ 2	1/ 2	1	1
3			2	1	0.92952E+02	0.92959E+02	0.73802E-02	0.73802E-02	0.36901E-02	49.87 %	49.87 %	37.6 %	37.6 %
4				2	0.92965E+02	0.91873E-02							
5	LAB. 2	2	1	1	0.93007E+02	0.93006E+02	0.92991E+02	0.92991E+02	0.29999E-01	0.11079E+02	0.11079E+02	0.29343E+01	1
6				2	0.93006E+02	0.70674E-03	0.18818E-01	0.18818E-01	0.20232E-01	1/ 2	1/ 2	1	1
7			2	1	0.92985E+02	0.92976E+02	0.90127E-02	0.90127E-02	0.14999E-01	92.04 %	92.04 %	91.3 %	91.3 %
8				2	0.92967E+02	0.12726E-01							
9	LAB. 3	3	1	1	0.92983E+02	0.92986E+02	0.92982E+02	0.92982E+02	0.74997E-02	0.50000E+01	0.50000E+01	0.29550E+00	1
10				2	0.92989E+02	0.42403E-02	0.51233E-02	0.51233E-02	0.47432E-02	1/ 2	1/ 2	1	1
11			2	1	0.92980E+02	0.92979E+02	0.33540E-02	0.33540E-02	0.37498E-02	84.52 %	84.52 %	41.3 %	41.3 %
12				2	0.92977E+02	0.21255E-02							
13	LAB. 4	4	1	1	0.92950E+02	0.92945E+02	0.92948E+02	0.92948E+02	0.50049E-02	0.50115E+00	0.50115E+00	0.12716E-05	1
14				2	0.92940E+02	0.70672E-02	0.64544E-02	0.64544E-02	0.00000E+00	1/ 2	1/ 2	1	1
15			2	1	0.92945E+02	0.92950E+02	0.70699E-02	0.70699E-02	0.35349E-02	44.76 %	44.76 %	1.0 %	1.0 %
16				2	0.92955E+02	0.70726E-02							
17	LAB. 5	5	1	1	0.92966E+02	0.92959E+02	0.92961E+02	0.92961E+02	0.29908E-02	0.17890E+00	0.17890E+00	0.16980E+01	1
18				2	0.92952E+02	0.98995E-02	0.60267E-02	0.60267E-02	0.00000E+00	1/ 2	1/ 2	1	1
19			2	1	0.92961E+02	0.92962E+02	0.70710E-02	0.70710E-02	0.35355E-02	28.65 %	28.65 %	80.8 %	80.8 %
20				2	0.92963E+02	0.14134E-02							

SAMPLE TYPE I U-235 RESULTS

TWO-WAY VARIANCE ANALYSIS

SYMBOL	VALUE	DOF	EXPLANATION
N	20	---	TOTAL NO. OF ITEMS
KT	10	---	TOTAL NO. OF GROUPS
R	5	---	NO. OF CLASSES
XGGG	0.92967E+02	---	OVERALL MEAN
STOT	0.19155E-01	19	OVERALL STD.DEV.
SIG	0.70275E-02	10	MEAN WITHIN GROUP STD.DEV.
SZG	0.14326E-01	5	MEAN BETWEEN GROUPS STD.DEV.
SW	0.36914E-01	4	BETWEEN CLASSES STD.DEV.
SB	0.88277E-02	5	BETWEEN GROUPS COMPONENT OF VARIABILITY
SA	0.17010E-01	4	BETWEEN CLASSES COMPONENT OF VARIABILITY
FB	0.41560E+01	5/ 10	SNEDECOR F-TEST ON SIGNIFICANCE OF SB
SLB	97.35 %	---	PROBABILITY LEVEL OF FB
FA	0.32727E+01	4/ 5	SNEDECOR F-TEST ON SIGNIFICANCE OF SA
SLA	88.72 %	---	PROBABILITY LEVEL OF FA
CHISQ1	0.69295E+01	9	BARTLETT TEST ON HOMOGENEITY OF GROUP VARIANCES
SLI	35.5 %	---	PROBABILITY LEVEL OF CHISQ1
CHISQ2	0.48515E+01	4	BARTLETT TEST ON HOMOGENEITY OF BETWEEN GROUP VARIANCES
SLZ	69.7 %	---	PROBABILITY LEVEL OF CHISQ2

LIST OF ITEMS: ONE-WAY VARIANCE ANALYSIS ON INDIVIDUAL SAMPLES

LINE NO.	SAMPLE CODE	CLASS NO.	GROUP NO.	ITEM NO.	ITEM VALUE	GROUP: MEAN STD.DEV.	CLASS: MEAN STD.DEV.	CLASS: SZ SH SE	CLASS: F DF1/DF2 SL	CLASS: CHISO DF SL
1	LAB. 1	1	1	1	0.93091E+02	0.93089E+02	0.93090E+02	0.14954E-02	0.21769E+00	0.32811E-01
2				2	0.93087E+02	0.28323E-02	0.27565E-02	0.00000E+00	1/ 2	1
3			2	1	0.93088E+02	0.93090E+02	0.32052E-02	0.16026E-02	31.33 %	14.4 %
4				2	0.93093E+02	0.35390E-02				
5	LAB. 2	2	1	1	0.93100E+02	0.93102E+02	0.93099E+02	0.55008E-02	0.26926E+01	0.29752E+00
6				2	0.93103E+02	0.21202E-02	0.41910E-02	0.30839E-02	1/ 2	1
7				1	0.93099E+02	0.93096E+02	0.33523E-02	0.27504E-02	75.75 %	41.5 %
8			2	0.93093E+02	0.42403E-02					
9	LAB. 3	3	1	1	0.93103E+02	0.93105E+02	0.93107E+02	0.45013E-02	0.32376E+01	0.55143E-01
10				2	0.93107E+02	0.28323E-02	0.33055E-02	0.26461E-02	1/ 2	1
11			2	1	0.93108E+02	0.93110E+02	0.25017E-02	0.22507E-02	78.62 %	18.6 %
12				2	0.93111E+02	0.21202E-02				
13	LAB. 4	4	1	1	0.93080E+02	0.93085E+02	0.93090E+02	0.10002E-01	0.20015E+01	0.12716E-05
14				2	0.93090E+02	0.70672E-02	0.81636E-02	0.50030E-02	1/ 2	1
15			2	1	0.93090E+02	0.93095E+02	0.70699E-02	0.50011E-02	70.72 %	1.0 %
16				2	0.93100E+02	0.70726E-02				
17	LAB. 5	5	1	1	0.93092E+02	0.93093E+02	0.93094E+02	0.24948E-02	0.49847E+01	0.29750E+00
18				2	0.93093E+02	0.70674E-03	0.17069E-02	0.15773E-02	1/ 2	1
19			2	1	0.93096E+02	0.93095E+02	0.11174E-02	0.12474E-02	84.48 %	41.5 %
20				2	0.93094E+02	0.14134E-02				

SAMPLE TYPE II U-235 RESULTS

ANNEX 3 (page 2b)

TWO-WAY VARIANCE ANALYSIS

SYMBOL	VALUE	DOF	EXPLANATION
N	20	---	TOTAL NO. OF ITEMS
KT	10	---	TOTAL NO. OF GROUPS
R	5	---	NO. OF CLASSES
XGGG	0.93096E+02	---	OVERALL MEAN
STOT	0.78599E-02	19	OVERALL STD.DEV.
SIG	0.39749E-02	10	MEAN WITHIN GROUP STD.DEV.
SZG	0.56396E-02	5	MEAN BETWEEN GROUPS STD.DEV.
SW	0.14634E-01	4	BETWEEN CLASSES STD.DEV.
SB	0.28288E-02	5	BETWEEN GROUPS COMPONENT OF VARIABILITY
SA	0.67518E-02	4	BETWEEN CLASSES COMPONENT OF VARIABILITY
FB	0.20129E+01	5/ 10	SNEDCOR F-TEST ON SIGNIFICANCE OF SB
SLB	83.79 %	---	PROBABILITY LEVEL OF FB
FA	0.29152E+01	4/ 5	SNEDCOR F-TEST ON SIGNIFICANCE OF SA
SLA	86.43 %	---	PROBABILITY LEVEL OF FA
CHISQ1	0.52623E+01	9	BARTLETT TEST ON HOMOGENEITY OF GROUP VARIANCES
SLI	18.9 %	---	PROBABILITY LEVEL OF CHISQ1
CHISQ2	0.26005E+01	4	BARTLETT TEST ON HOMOGENEITY OF BETWEEN GROUP VARIANCES
SLZ	37.3 %	---	PROBABILITY LEVEL OF CHISQ2



## SAMPLE TYPE III

## U-235 RESULTS

ANNEX 3 (page 3a)

## LIST OF ITEMS: ONE-WAY VARIANCE ANALYSIS ON INDIVIDUAL SAMPLES

LINE NO.	SAMPLE CODE	CLASS NO.	GROUP NO.	ITEM NO.	ITEM VALUE	GROUP MEAN	STD.DEV.	CLASS MEAN	STD.DEV.	CLASS SZ	CLASS SH	CLASS SE	CLASS F	CLASS DF1/DF2	CLASS CHISQ
1	LAB. 1	1	1	1	0.93268E+02	0.93270E+02	0.93266E+02	0.93266E+02	0.84991E-02	0.11542E+02	0.55143E-01				
2				2	0.93272E+02	0.28323E-02	0.53151E-02	0.57436E-02		1/ 2	1				
3			2	1	0.93260E+02	0.93262E+02	0.25017E-02	0.42496E-02		92.32 %	18.6 %				
4				2	0.93263E+02	0.21202E-02									
5	LAB. 2	2	1	1	0.93273E+02	0.93275E+02	0.93275E+02	0.99948E-03	0.68969E-01	0.43036E+00					
6				2	0.93276E+02	0.21202E-02	0.31605E-02	0.00000E+00		1/ 2	1				
7			2	1	0.93272E+02	0.93276E+02	0.38058E-02	0.19029E-02		18.26 %	48.8 %				
8				2	0.93279E+02	0.49470E-02									
9	LAB. 3	3	1	1	0.93272E+02	0.93273E+02	0.93273E+02	0.49597E-03	0.26556E-01	0.15029E+01					
10				2	0.93273E+02	0.70674E-03	0.25014E-02	0.00000E+00		1/ 2	1				
11			2	1	0.93270E+02	0.93273E+02	0.30435E-02	0.15217E-02		11.45 %	78.0 %				
12				2	0.93276E+02	0.42457E-02									
13	LAB. 4	4	1	1	0.93275E+02	0.93270E+02	0.93265E+02	0.10002E-01	0.20000E+01	0.00000E+00					
14				2	0.93265E+02	0.70726E-02	0.81667E-02	0.50011E-02		1/ 2	1				
15			2	1	0.93255E+02	0.93260E+02	0.70726E-02	0.50011E-02		70.71 %	1.0 %				
16				2	0.93265E+02	0.70726E-02									
17	LAB. 5	5	1	1	0.93273E+02	0.93272E+02	0.93269E+02	0.60043E-02	0.72183E+01	0.29752E+00					
18				2	0.93271E+02	0.14134E-02	0.39175E-02	0.39407E-02		1/ 2	1				
19			2	1	0.93268E+02	0.93266E+02	0.22348E-02	0.30022E-02		88.49 %	41.5 %				
20				2	0.93264E+02	0.28269E-02									

## SAMPLE TYPE III

## U-235 RESULTS

ANNEX 3 (page 3b)

## TWO-WAY VARIANCE ANALYSIS

SYMBOL	VALUE	DOF	EXPLANATION
N	20	---	TOTAL NO. OF ITEMS
KT	10	---	TOTAL NO. OF GROUPS
R	5	---	NO. OF CLASSES
XQQQ	0.93270E+02	---	OVERALL MEAN
STOT	0.59877E-02	19	OVERALL STD.DEV.
SIG	0.41236E-02	10	MEAN WITHIN GROUP STD.DEV.
SZQ	0.64742E-02	5	MEAN BETWEEN GROUPS STD.DEV.
SW	0.86772E-02	4	BETWEEN CLASSES STD.DEV.
SB	0.35292E-02	5	BETWEEN GROUPS COMPONENT OF VARIABILITY
SA	0.28887E-02	4	BETWEEN CLASSES COMPONENT OF VARIABILITY
FB	0.24650E+01	5/ 10	SNEDECOR F-TEST ON SIGNIFICANCE OF SB
SLB	89.47 %	---	PROBABILITY LEVEL OF FB
FA	0.12833E+01	4/ 5	SNEDECOR F-TEST ON SIGNIFICANCE OF SA
SLA	61.24 %	---	PROBABILITY LEVEL OF FA
CHISQ1	0.53093E+01	9	BARTLETT TEST ON HOMOGENEITY OF GROUP VARIANCES
SLI	19.3 %	---	PROBABILITY LEVEL OF CHISQ1
CHISQ2	0.54366E+01	4	BARTLETT TEST ON HOMOGENEITY OF BETWEEN GROUP VARIANCES
SLZ	75.5 %	---	PROBABILITY LEVEL OF CHISQ2